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Ultrafiltration of sugarcane juice with spiral wound modules: on-site pilot trials

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Abstract

This work investigates the performance of spiral wound membrane modules for the ultrafiltration (UF) of sugarcane juice. In on-site trials carried out in an Indian sugar mill, different modules with varying channel sizes employing 20 kDa polyethersulphone and polysulphone membranes were evaluated. Two juice streams viz. raw juice and clarified juice were selected in the sugar manufacturing process and the effect of such operating parameters as transmembrane pressure, operating time and feed temperature on the permeate flux and quality were examined. It was observed that a narrow channel size resulted in a higher flux with a corresponding lower membrane fouling. The flux could be further improved by increasing the operating temperature. With both the feed streams, the permeate was observed to be consistently superior in terms of higher clarity, lower color and reduced calcium oxide content as compared to the clarified juice produced by the conventional double sulphitation process. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Sugarcane juice; Ultrafiltration; Spiral wound module; Flux; Permeate

1. Introduction

The purification of sugarcane juice by membrane filtration promises a significant improvement in the sugar quality and yield [1]. In spite of extensive laboratory trials [2–8], field tests aimed at full-scale operation have been conducted only in the last decade [9–12]. India represents a significant market for this technology since it is the world's largest producer of plantation white sugar by the double sulphitation process. The manufacturing scheme involves treating the sugarcane juice with lime and sulphur dioxide, followed by heating above 100°C. The sludge thus formed is separated by settling and filtration. Owing to the presence of non-sugar impurities like dextrans and waxes in the colloidal form, the dark yellowish brown clarified juice is typically cloudy. This has an adverse effect upon the color of the sugar produced. There is thus an urgent need in the sugar industry for an effective clarification process that can consistently produce clear juice, preferably with reduced color.

The present investigation explores the performance of polymeric spiral wound modules for cane juice ultrafiltration (UF) in a sugar mill. Keeping in view the large membrane area required for an eventual full-scale application, the emphasis was on the evaluation of low cost modules manufactured indigenously.

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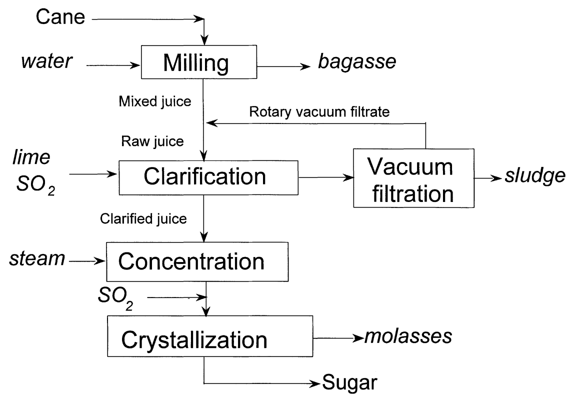

Fig. 1. Schematic of plantation white sugar manufacture.

Two feed streams in the manufacturing process (Fig. 1) viz. raw juice and clarified juice were studied. The effects of module channel height, operating pressure, operating time and feed temperature on juice flux and purity were examined. This data is expected to aid in the subsequent design of an UF demonstration plant with a juice handling capacity of 10 m^3/h.

2. Experimental

2.1. Membranes

Table 1 lists the spiral wound membrane modules that were evaluated for the treatment of sugarcane juice. The Cellpore® module was supplied by Inchema Consulting AG, Zurich, Switzerland. The remaining modules were obtained from Permionics, Vadodara, India. All the modules were commercially available and used without further modification except where specifically mentioned otherwise.

2.2. Ultrafiltration (UF) runs

The trials were performed on-site at the Simbhaoli Sugar Mills Ltd. which is located about 100 km from Delhi at Simbhaoli village, Ghaziabad district (Uttar Pradesh). The investigations spanned two cane crushing seasons between November 1997 and May 1999.

All experiments were conducted with fresh juice collected from the selected stage in the sugar manufacturing process. The feed was pre-treated appropriately and thereafter filtered through a combination of stainless steel (SS) screens to remove suspended particles. The pre-filtered feed (80–100 l) was then pumped through a fixed speed plunger pump (American Spring and Pressing Works Ltd., Mumbai, India) to the single spiral element. Depending upon the channel size, the effective filtration area varied in the 1–2.2 m^2 range. No temperature control was attempted for the feed though the feed temperature was monitored over the course of the experiments.

The UF was typically performed in a batch mode either with full retentate recycle or with a recirculating loop accompanied by periodic feed topping. Selected runs were conducted in the feed and bleed mode. At the end of each experiment, the membranes were thoroughly flushed with condensed steam (pan condensate) diluted with raw water. The membrane fouling, expressed as a percentage drop in the water permeability, was estimated by measuring the pure water flux both before and after the cane juice UF. The membranes were then cleaned in place using

Table 1
Membrane module details

Membrane module[a]	Manufacturer	Membrane material	Module dimensions (cm×cm)[b]	Effective filtration area (m^2)	PWP[c] (LMH per kg/cm^2)
PPE0106 (40 mil)	Permionics, India	Polyethersulphone	6.35×102	2.2	58.0
PPE0106 (60 mil)	Permionics, India	Polyethersulphone	6.35×81	1.0	12.5[d]
PPE0106 (80 mil)	Permionics, India	Polyethersulphone	6.35×93	1.4	29.1, 33.87[e]
Cellpore® (0.8 mm)	Inchema, Switzerland	Modified polysulphone	8×47	1.3	41.70

[a] Nominal molecular weight cutoff 20 kDa.
[b] Diameter×length.
[c] PWP experimental values, obtained at ambient temperature (28–30°C) with new modules.
[d] Used module, cleaned before PWP measurement.
[e] Different batches of modules.

the appropriate cleaning solution before storing in 0.5–1% formalin till the subsequent run.

2.3. Juice analysis

The juice pH was measured using a calibrated pH meter with automatic temperature compensation (Control Dynamics, India). The juice color and clarity were estimated at 580 nm with a spectrophotometer (Systronics, India). The brix, representing the total dissolved solids, was measured using a standardized brix spindle (0–10 or 10–20 brix range, Reige, Germany). The 'pol' (a measure of the total polarizing substances, primarily sucrose), was estimated using a polarimeter (Schmitz and Heinsch, Germany). The purity rise and the rejection of various juice components was calculated as follows:

$$\text{Purity (\%)} = \left(\frac{\text{Pol per cent juice}}{\text{Brix}}\right) \times 100$$

$$\text{Purity rise} = (\text{Purity})_{\text{permeate}} - (\text{Purity})_{\text{feed}}$$

$$\text{Brix rejection (\%)} = \left(1 - \frac{(\text{Brix})_{\text{permeate}}}{(\text{Brix})_{\text{feed}}}\right) \times 100$$

$$\text{Non-sugars rejection (\%)} = \left(1 - \frac{(\text{Brix} - \text{Pol})_{\text{permeate}}}{(\text{Brix} - \text{Pol})_{\text{feed}}}\right) \times 100$$

$$\text{Sugar rejection (\%)} = \left(1 - \frac{(\text{Pol})_{\text{permeate}}}{(\text{Pol})_{\text{feed}}}\right) \times 100$$

The calcium oxide (CaO) content was estimated by titration. All analytical methods were as per the mill's practice and were in accordance with the norms prescribed by the Sugar Technologists Association of India [13].

3. Results

Two different juice streams viz. raw juice and clarified juice were evaluated on single spiral elements. The raw juice, tapped from the raw juice heaters at 70°C, is a turbid, dark greyish green solution at a pH between 5.7 and 5.9. It has a high suspended solids content of 15–25 g/l. In the conventional clarification process, the raw juice is limed, sulphited, boiled and subsequently allowed to settle in a clarifier. The overflow from the clarifier constitutes the clarified juice stream which is then taken to the evaporators for concentration. The clarified juice at 102°C is typically yellowish brown with a pH in the 6.95–7.05 range. Though this juice is normally free from visible suspended particles, it is generally hazy due to the presence of colloidal matter and fine bagasse particles (bagacillo).

3.1. Raw juice

The experiments were conducted on heated raw juice that was limed, flocculated and allowed to settle for 20–30 min to remove the suspended solids [14]. The clear juice obtained from this step was prefiltered through SS filters (60 mesh followed by 120 mesh) before subjecting it to UF. The experiments with the Permionics spiral modules were conducted after hydrophilizing the polyethersulphone membrane surface by the adsorption of polyvinyl alcohol (PVA). The procedure involved circulating a 0.1% PVA solution over the membrane surface for approximately 5 min. The module was then flushed with water to remove the excess, unadsorbed PVA before conducting the juice UF. The membrane surface modification was performed since previous field trials on a crossflow module consistently exhibited a higher flux with the PVA adsorbed membrane [15]. Further, the corresponding membrane fouling was also lower (60% drop in pure water permeability (PWP) with surface modification as compared to 73.4% drop with the unmodified membrane).

Fig. 2 shows the effect of varying transmembrane pressure on the raw juice flux through different spiral modules. The pressure drop was 1.6–2 kg/cm^2 for both the Permionics modules and 1.1–2 kg/cm^2 for the Cellpore module. The flux data reported for each pressure is averaged over the first 10 min of operation once the pressure was stable (typically within 1–2 min). The 40 mil spiral from Permionics exhibited the highest flux of about 58 LMH at 2 kg/cm^2. The flux was twice that obtained with the 80 mil Permionics modules (29.4 LMH). A Cellpore® 32 mil module, which was tested for comparison, exhibited a flux of 30.2 LMH. Further, with both Permionics 80 mil and Cellpore® 32 mil modules, the flux plateau was reached earlier at 1 kg/cm^2. It was also observed that in the mass transfer controlled region, the Permionics spirals displayed a marginal flux decline with increasing pressure beyond 3 kg/cm^2.

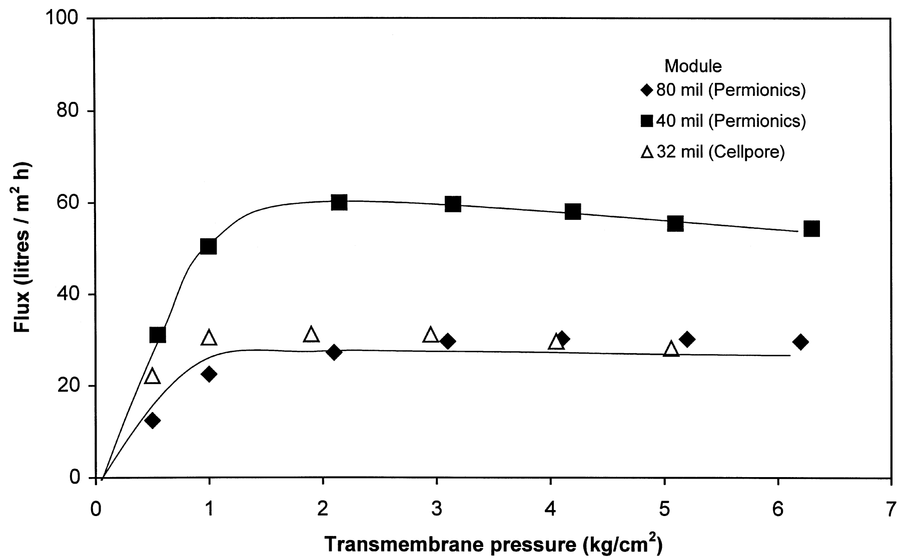

Fig. 2. Raw juice: effect of TMP (feed flow rate 24 l/min; pH 7.06–7.09; temperature 50–53°C).

The raw juice has a high suspended solids content mainly in the form of bagacillo. Earlier experiments have demonstrated that the fine particulate matter tends to deposit on the membrane surface during the course of raw juice UF [16]. The deposits form a secondary filtration layer that gets compressed at higher applied pressures. This increases the resistance to permeate flow and thus leads to flux decline with increasing pressure. Similar behavior has also been reported with 0.05 μm tubular ceramic membranes processing sugarcane juice at pressures of 2–23 kg/cm^2 and recirculation velocities of 1–5.5 m/s [7] as well as with other highly fouling liquid streams like acid whey and skim milk [16]. It was observed in our studies that the initial pre-treatment step involving flocculation aided in the formation of large flocs that could be eliminated by subsequent settling. However, complete removal of the bagacillo fines could not be effected even when the juice was filtered twice through 150 mesh SS sieves.

A comparison of the Permionics modules indicates that the flux improves with reduced channel size (Figs. 2 and 3). This is explained by the reasoning that a narrow channel results in a higher crossflow velocity (0.20 m/s for the 40 mil module as compared to 0.10 m/s for the 80 mil module). This, in turn, leads to better concentration polarization control in the 40 mil element. However, the behavior of the 32 mil Cellpore module was almost identical to that of the 80 mil Permionics module in spite of the difference in channel height. This could be caused by the higher fouling (69% reduction in water permeability) of the modified polysulphone membrane employed in the Cellpore module. On the contrary, the PVA adsorbed polyethersulphone membrane in the 80 mil Permionics spiral exhibited comparatively less fouling (45% drop in PWP).

The effect of channel height becomes particularly significant during long-term operation. Fig. 4 exhibits the UF of pre-treated raw juice on a Permionics 80 mil module. The experiment was conducted in a batch mode with retentate recycle accompanied by periodic feed topping. Once the feed volume was reduced by 20% (volume concentration factor 1.25X), the feed tank was replenished with fresh pre-treated raw juice equal to the volume of permeate withdrawn. Since cane juice is extremely prone to microbial degradation as well as sugar loss due to inversion with increased processing time, the feed was changed at regular intervals as marked in Fig. 4. After every 2–3 h of operation, the module was drained completely of the old feed and the experiment was continued with a fresh batch of feed, treated appropriately. No module cleaning was performed between feed changes.

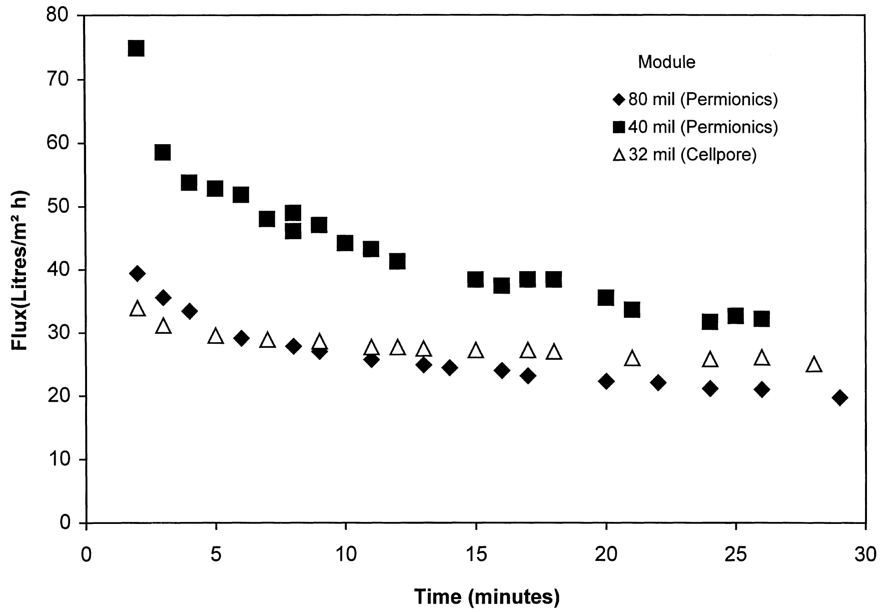

Fig. 3. Raw juice: flux vs. operating time (feed flow rate 24 l/min; TMP 6.1–6.2 kg/cm^2; pH 7.02–7.22; temperature 50–58°C).

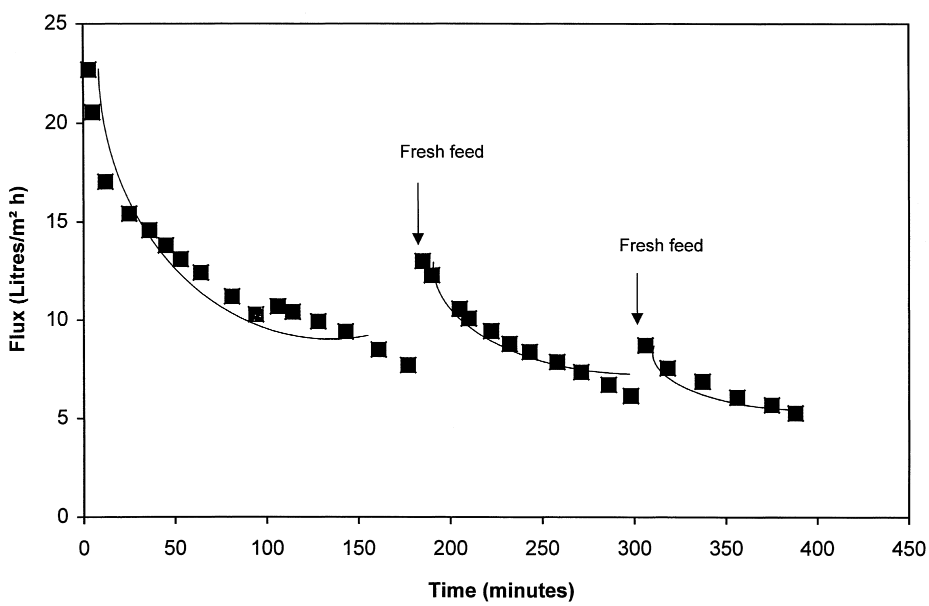

Fig. 4. Raw juice: batch mode with periodic feed topping (Permionics 80 mil module; feed flow rate 24 l/min; pH 7.03–7.57; TMP 6.38 kg/cm^2; temperature 50°C).

It was observed that the flux displayed an exponential decrease with time. However, when the experiment was resumed with fresh feed, there was an initial rise in the flux but it declined exponentially thereafter. Over a period of nearly 7 h, the flux dropped to ∼5 LMH. The decline is possibly aggravated by the high TMP (6.2 kg/cm^2) during the course of this run. Further, the rejected matter on the membrane surface acts as a secondary filtration element. At higher applied pressure, the layer compaction is more pronounced with increasing operating time and so the initial higher flux due to the larger driving force gets offset by the flux drop due to the increased resistance offered by the compressed secondary layer. This result yet again points out that polarization control may not be very effective in larger channels. Thus, to ensure acceptable fluxes during continuous operation, a combination of narrow channel height and low operating pressure would have to be chosen.

To simulate continuous operation, the raw juice UF was performed in the feed and bleed mode on a Permionics 40 mil module (Fig. 5). An average flux of 40.9 LMH was obtained at 58°C with a transmembrane pressure of 5.1 kg/cm^2. It was observed that the flux was stable during the course of the experiment lasting 2 h. The percent volume reduction was 72% (concentration factor 3.6X) and the average purity rise with respect to the feed was 1.48 units.

In all the experiments with raw juice, the UF permeate was observed to be sparkling clear. Table 2 presents a comparison of the permeate characteristics. It was observed that the average purity rise across UF was over 2 units. This is a significant improvement over the 0.5–1.0 unit rise which is typically obtained in conventional clarification. However, the permeate color varied with the module type. The Permionics modules produced a visibly lighter, golden yellow colored permeate which was superior to that of the Cellpore module. This observation was supported by the higher transmittance values of the permeate obtained with the Permionics spirals. Further, it was observed that the CaO content of the ultrafiltered juice was consistently lower than that of the conventional clear juice. This is an additional benefit since a lower CaO content would reduce the extent of evaporator fouling and consequently reduce the downtime for cleaning as well as the consumption of cleaning chemicals.

Of the three modules tested, the Permionics spiral with the 40 mil channel exhibits the best combination of high flux and high purity rise. However, this mod-

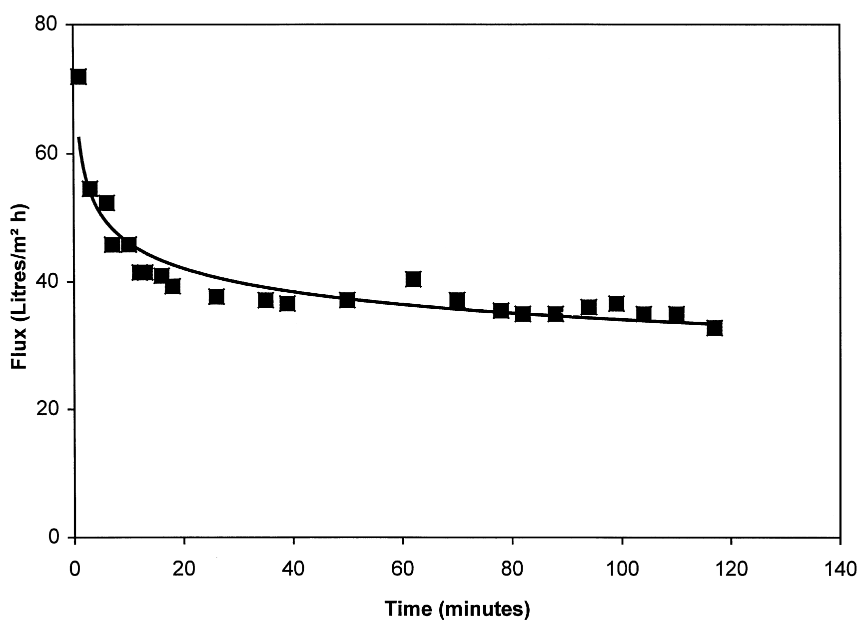

Fig. 5. Raw juice: feed and bleed mode (Permionics 40 mil module; feed flow rate 24 l/min; pH 7.0–7.2; TMP 5.1 kg/cm^2; temperature 58°C).

Table 2
Permeate characteristics: raw juice feed

Property	Conventional clarification	Ultrafiltration		
		Permionics		Cellpore
		40 mil	80 mil	32 mil
Color	Yellowish brown	Golden yellow	Golden yellow	Dark yellowish brown
Average purity rise	0.5–1.0	2.2	2.5	2.4
CaO content (mg/l)	1300–1400	<1000	950–970	900–1010
Absorbance	0.506	<0.04	<0.04	>0.3
Transmittance (%)	31.2	>90	>90	<50
Brix rejection (%)	–	11.08	7.61	8.52
Non-sugars rejection (%)	–	18.21	18.13	22.35
Sugar rejection (%)	–	9.56	5.15	5.26

ule is also characterized by a relatively high sugar rejection (9.56%). This aspect needs to be investigated further to minimize the sugar retention in the UF process.

Table 3 exhibits the fouling of the membrane modules after raw juice UF. The PVA adsorbed polyethersulphone based 40 mil Permionics module displayed the minimum fouling while the modified polysulphone based Cellpore spiral showed the highest drop in PWP after juice UF. The raw juice is a strong foulant because of the presence of objectionable amounts of proteins, polysaccharides, starches, gums and other non-sugars that constitute up to 2.5% of the juice [17]. Table 4 describes the cleaning procedure investigated for restoring the original PWP of the membrane modules. A combination of 0.5% HCl wash followed by 0.1% caustic at 50°C led to 60–80% recovery of the clean membrane PWP. The membrane washing was performed throughout either with raw water or with condensed steam diluted with raw water. It was observed that the raw water displayed a high conductivity (>400 μS) due to the presence of dissolved salts. It is expected that the cleaning would be more effective if the membrane washing is performed using demineralized water.

Table 3
Fouling of membrane modules: raw juice feed

Module	PWP (LMH per kg/cm^2)		Fouling (%)
	Clean membrane	Fouled membrane	
Permionics (40 mil)	40.72[a]	35.37	13.14
Permionics (80 mil)	18.48[a]	10.17	44.97
Cellpore® (32 mil)	41.70	12.98	68.87

[a] Clean membrane, after PVA adsorption.

3.2. *Clarified juice*

The clarified juice was tapped from the clarifier outlet that is located before the juice heaters upstream to the evaporator inlet. A combination of 60 mesh and 120 mesh SS sieves was employed to pre-filter the clarified juice to eliminate any suspended particles (bagacillo) from the feed stream.

Fig. 6 compares the effect of operating pressure on juice flux for the 60 and 80 mil channel Permionics modules. The experiment was performed in increasing (filled symbols) and decreasing (open symbols) pressure modes. The pressure drop was 1 kg/cm^2 for both the modules. The flux through the 80 mil module reached a maximum at 3 kg/cm^2 after which there was a gradual decline at higher pressures. On reversing the pressure at 6.5 kg/cm^2, the course of the curve shifted downwards. A pronounced hysteresis was observed in this case. In contrast, the 60 mil module displayed an almost linear increase in the flux with increasing TMP till 5.5 kg/cm^2. This trend was independent of the increasing or decreasing mode of operation.

A similar hysteresis effect has been reported by Kishihara et al. [3] in stirred cell experiments at 60°C on mixed juice limed to pH 8.0 using Amicon XM300A membranes. They noticed that the flux increased with increasing pressure up to 2 kg/cm^2 but decreased thereafter. On gradually releasing the pressure from 4 kg/cm^2, the flux decreased through

Table 4
Cleaning procedure for Permionics spirals

Sequence of operations	Permeability (LMH per kg/cm^2)	Recovery (%)
Procedure 1		
Water flux of clean membrane	44.6	
Water flux of fouled membrane	9.96	
Water flux after 5 min acid (0.5% HCl) wash at ambient temperature[a]	22.71	50.92
Water flux after 10–15 min 0.1% caustic wash at 50°C	27.77	62.26
Water flux after 15 min 0.1% caustic wash at 60°C	34.35	77.02
Procedure 2		
Water flux of fouled membrane at ambient temperature[a]	19.64	
Water flux after 10 min hot water (65°C) wash	23.64	53.00
Water flux after 5 min acid (0.5% HCl) wash	19.09	42.80
Water flux after 0.1% NaOH wash at ambient temperature[a]	26.48	59.37

[a] Ambient temperature was ∼32°C.

a different course than that followed during the increase in pressure. The explanation was that a compact gel layer was formed which loosened somewhat with the release of pressure but still remained intact on the membrane surface. This behavior was more prominent with the high flux XM300A membrane as compared to the tighter PM10 membrane.

In our studies, the hysteresis effect was noticeable with the 20 kDa polyethersulphone membrane in the 80 mil module. The same membrane in a 60 mil element did not display this behavior. This result once again emphasizes the choice of optimum spacers in spiral elements. A narrow channel would lead to higher feed velocity on the membrane surface (0.14 m/s with a 60 mil spacer as opposed to 0.10 m/s with a 80 mil spacer). This would reduce the extent of concentration polarization due to the layer formed by the rejected macromolecular impurities and thus the flux is likely to increase proportionately with increasing pressure as observed with the 60 mil module.

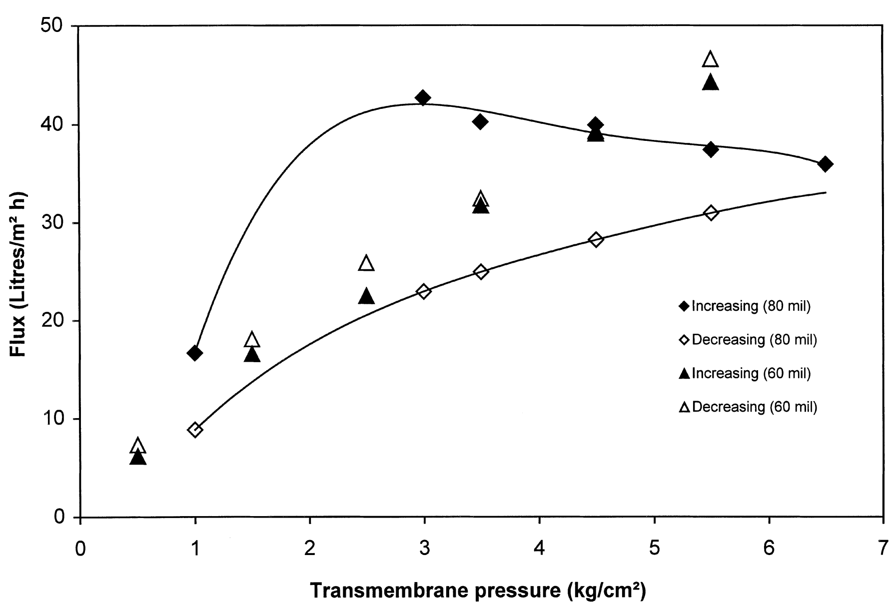

Fig. 6. Clarified juice: effect of TMP (feed flow rate 24 l/min; temperature 53–64°C).

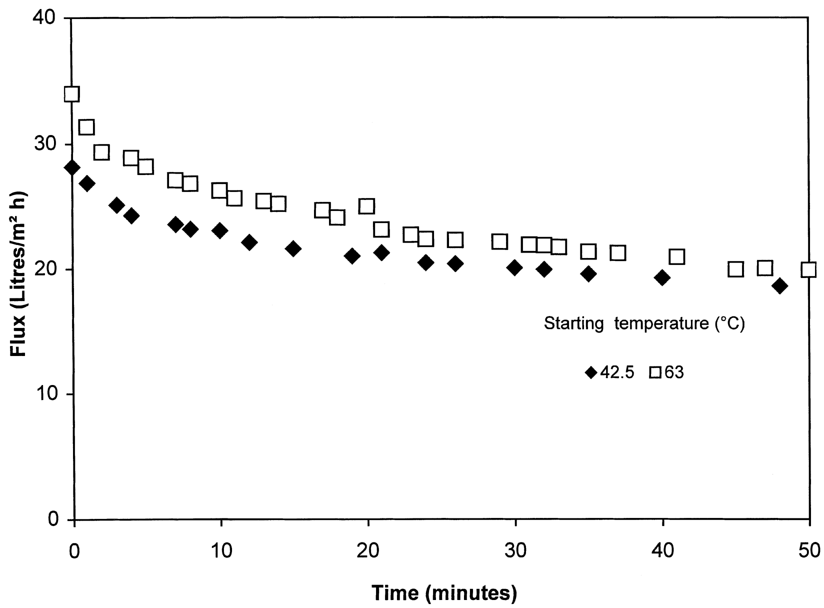

Fig. 7. Clarified juice: effect of temperature (Permionics 80 mil module; feed flow rate 24 l/min; TMP 3.5 kg/cm^2).

Fig. 7 displays the effect of temperature on clarified juice UF. Experiments were conducted in the retentate recycle mode with a concentration factor of 4.3X. In agreement with reported literature [4], the flux is higher at a higher temperature. However, the benefits of a higher starting temperature were lost since no temperature control was attempted. This resulted in a gradual cooling of the feed as the experiment progressed. For instance, with a starting temperature of 63°C, it was observed that the juice temperature dropped to 50°C in 1 h of operation. The difference in the average juice fluxes was, therefore, marginal (22.1 and 24.4 LMH at starting temperatures of 42 and 63°C, respectively).

To simulate continuous operation, the UF was performed in a feed and bleed mode (Fig. 8) on a Permionics 80 mil spiral element. Fresh feed was introduced into the loop at about the same rate as the permeate plus bleed flow rates. The initial juice flux was high, possibly because the starting temperature was high (nearly 80°C). However, in the absence of any feed heating, the juice cooled rapidly and the average operating temperature during the course of the run was 60°C. An average flux of 16 LMH was obtained with 90% volume recovery (concentration factor 10X). A similar run on the 60 mil module exhibited over twice the flux with an average value of 35 LMH (Fig. 9). The operating temperature in this run was 67°C and the UF was conducted at 93% recovery (concentration factor 14X).

Our results with the 60 mil module compare well with the only other reported field test on sugarcane juice using spiral wound modules. In pilot trials carried out with 50 kDa polyvinyledene diflouride (PVDF) spiral elements supplied by Koch Membrane Systems, fluxes of 60–80 LMH were reported at an operating temperature of 95–98°C [12].

Table 5 lists the permeate characteristics of clarified juice. With both the modules, a clear golden filtrate was obtained. The Permionics 60 mil module was observed to be better in terms of higher flux combined with higher purity rise. However, the sugar rejection was also high at 9% with this element. A similar trend was observed with the raw juice feed with decreasing channel height leading to higher sugar rejection. This aspect needs further investigation.

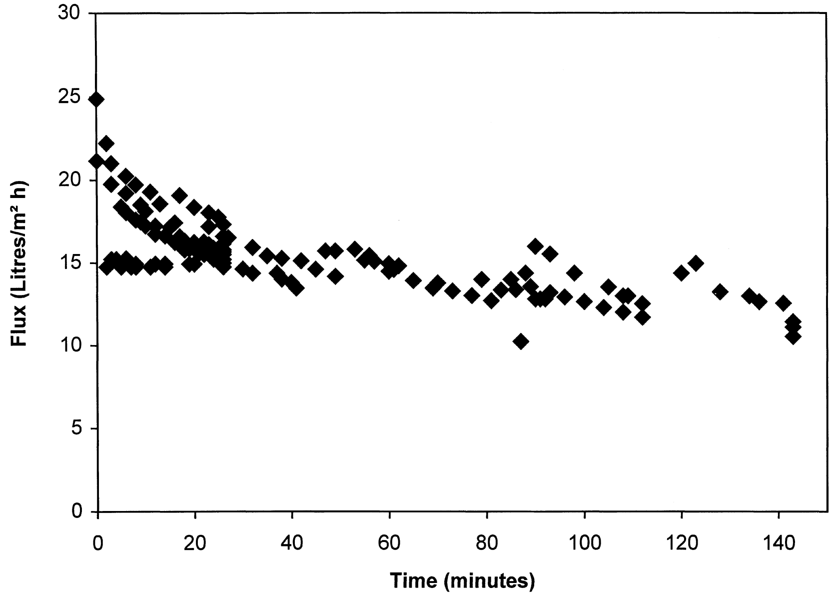

Fig. 8. Clarified juice: feed and bleed mode (Permionics 80 mil module; feed flow rate 24 l/min; TMP 3.5 kg/cm^2; average temperature 60°C).

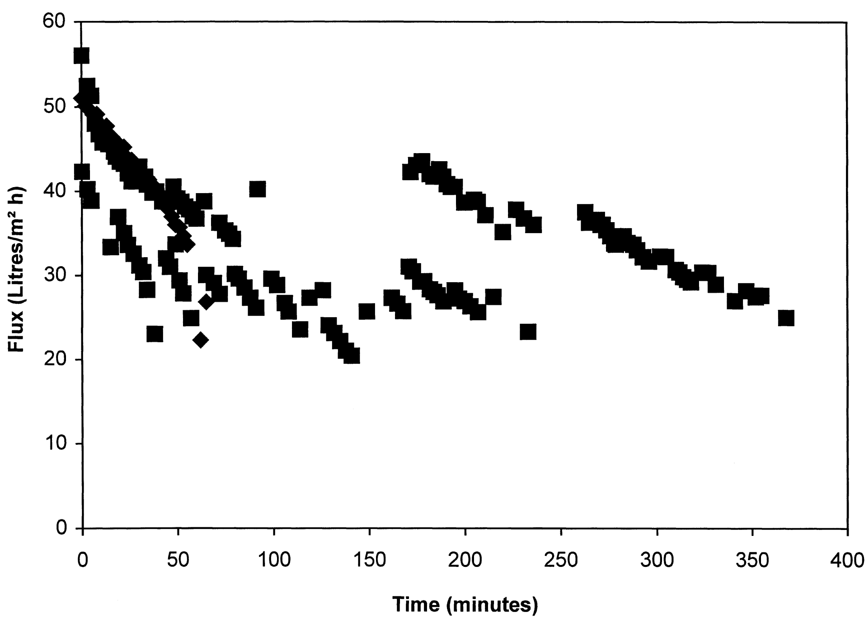

Fig. 9. Clarified juice: feed and bleed mode (Permionics 60 mil module; feed flow rate 24 l/min; TMP 3.5 kg/cm^2; average temperature 67°C).

Table 5
Permeate characteristics: clarified juice feed

Property	Permionics	
	60 mil	80 mil
Average flux[a] (LMH)	31.53	21.64
Purity rise	2.10	1.48
Brix rejection (%)	11.47	4.64
Non-sugars rejection (%)	19.01	11.05
Sugar rejection (%)	9.00	2.91

[a] Averaged over five experiments.

4. Conclusions

This work presents an on-site assessment of polymeric spiral wound modules for sugarcane juice UF. The variation in flux and juice properties with varying parameters like module channel height, transmembrane pressure and feed temperature were investigated on the raw juice and clarified juice streams. The main points that emerged from this study are listed below:

- UF produces a permeate that is consistently superior in terms of lower color, higher clarity and lower CaO content as compared to the clarified juice obtained by the conventional liming-sulphitation process.
- The average purity rise across UF was greater than 2 units with raw juice feed and over 1.5 units with clarified juice feed. These values are a significant improvement over the 0.5–1 unit purity rise obtained in the conventional clarification scheme.
- Both the raw juice and clarified juice streams were characterized by the presence of bagacillo fines which could not be eliminated by pre-filtration through sieves up to 150 mesh. These fines have a tendency to deposit upon the membrane surface during the course of UF thus creating a secondary layer that eventually controls the juice filtration characteristics.
- A combination of narrow channel height at low operating pressure is likely to result in a satisfactory flux, particularly during long-term operation.
- Among the modules tested, the Permionics spirals with low channel heights (40 or 60 mil channel spacers) appear to be the best choice in terms of both higher flux and improvement in juice color.

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